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13. ABSTRACT (Maximum 200 words)

The overall goals of this work were to relate the microstructure, including the defect structure and impurity content and location, to the mechanical and physical properties of ice.

Research focused on determining the structure and microstructural location of impurities in natural ice using a scanning electron microscope equipped with a cold stage and x-ray microanalysis. Thus, for the first time, the microstructure and microchemistry of pond and river ice was characterized at high resolution (<1 μm). Preliminary mechanical testing was performed on the pond ice.

Preliminary electrical measurements were performed on sulfuric acid-doped ice, which demonstrate dramatic differences in the resistivity of the lattice and grain boundaries. Equal channel angular extrusion was used to examine the effects of impurities on the recrystallization of single crystal ice. A scanning electron microscope and a cold-stage were used to examine the structure of uncoated snow.

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The Micromechanisms of Flow and Fracture of Ice
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Goals

The long-term goals of this work are to:

1. Provide a detailed characterization of the microstructure as basis for further modeling of the mechanical behavior of ice in both ductile and brittle regimes;
2. To investigate the effect of impurities on the mechanical properties of ice.

Objectives

The near-term goals of this project were to:

1. Perform microstructural characterization of impurities (type and location) in seasonal ice using scanning electron microscopy coupled with x-ray microanalysis;
2. Investigate the brittle compressive strength of seasonal pond ice;
3. Determine how impurities affect the mechanical properties of ice;
4. Investigate the effect of impurities on the recrystallized microstructure of highly deformed ice;
5. Qualitatively assess the impurity content using electrical measurements.

Approach

A controlled-sublimation technique using a Princeton-Gamma-tech Energy Dispersive X-ray Spectroscopy, EDS, system attached to a low-vacuum scanning electron microscope, LVSEM, (both purchased with ARO funds) equipped with a custom built

cold-stage was used to determine the structure and the microstructural location of impurities in seasonal ice and to examine snow.

A custom-built straining jig, housed in a double-walled box with carefully controlled temperature, and controlled by a computer was used for the study of the effects of sulfuric acid on the creep of ice. Data was acquired using a second computer equipped with an in-house-built acquisition system.

A 120° Equal Channel Angular Extrusion jig, ECAE, was built and utilized to investigate the effect of sulfuric acid on recrystallization and subsequent grain boundary mobility in ice.

The brittle strength of seasonal ice (pond ice) was investigated by performing uniaxial and biaxial compression experiments using the MTS tri-axial servo-hydraulic testing system housed in cold-room in the Ice Research Laboratory at Dartmouth College.

Tasks Completed

We completed preliminary examinations of the microstructure and microchemistry of pond and river ice at high resolution (<1 μm) using LVSEM and EDS.

The brittle compressive strength of accretion pond ice was investigated by performing a series of uniaxial and biaxial compression experiments at –10°C using a triaxial MTS servo-hydraulic testing system.

The effects of impurities on the creep and primary recrystallization of ice was studied.

Preliminary electrical measurements demonstrated that the d.c. electrical conductivity of grain boundaries in sulfuric acid-doped ice is significantly higher than that of the bulk.

Lastly, a technique that does not require the prior application of a conductive metal coating was developed for the study of snow using a LVSEM. The method has the advantages of being simple and inexpensive in the context of providing a reasonable level of detail comparable in some cases to coated snow crystals. Moreover, the technique allows the use of EDS, as well as real-time observations of a variety of sublimation-induced morphological changes in the snow-aggregates.

Results

Microstructural characterization of impurities in seasonal ice

Samples were produced from cores harvested during January and February 2001, February 2002, and February and March 2003 from the Lower Baker Pond (Wentworth-Orford, NH). Ice from the Connecticut River (Hanover, NH) was also collected in February 2001. Structurally, both pond and river accretion ice were characterized by relatively large columnar grains whose diameters increase significantly as depth increases. Some microstructures at the top of the ice sheet were comprised almost entirely of very large grains (>70-80 mm) while some exhibited relatively smaller grains (15-30 mm) or a mixture of smaller grains and very large grains. The distinctive features of the top microstructures were the straight and in some instances quite long grain boundaries (>70-80 mm), Figure 1. This aspect is important because such boundaries are weak interfaces that can easily decohere under thermal or mechanically induced stresses leading to relatively large crack precursors with dramatic effects on the strength of the ice (more below). The decohesion of the boundaries is expected to occur at even lower applied stresses if impurities are present in the grain boundaries.

Thin-sections from the bottom of the ice sheet revealed the presence of rather convoluted and windy grain boundaries. Grains had a variety of shapes and in general along a certain direction they exceeded 100 mm.

SEM/EDS analysis of pond ice revealed that impurities were mostly localized in unexpected quasi-round crater-type features, and/or clusters located mainly on grain and at triple junctions, but occasionally in the lattice. The typical diameter of these features ranged between 0.2 mm and 1.2 mm. EDS analysis revealed, besides oxygen (from water), the presence of sodium and potassium, calcium and magnesium, chlorine, sulfur, as well as silicon and carbon. Because these impurities originate presumably from the weathering of various silicate minerals that compose the bedrock of the region, from the decomposition organisms in the sediments lying on the bottom of the pond (organic acids) as well as from anthropogenic sources, they were encountered at all locations, all depths, and in ice from all three years. Notable is the fact that qualitatively the density of

the impurity features was significantly lower in the 2002 ice compared to 2001 ice most likely due to the much colder 2001-2002 winter.

In river ice, impurity-aggregates were significantly smaller than the crater-like features or clusters observed in pond ice and were located only within or in the immediate vicinity of grain boundaries. Arrows in Figure 2a point to impurity-clusters. In general, the chemical composition of the impurities in pond ice and river ice was similar except for aluminum and iron (in low concentrations) which were observed only in river ice, Figure 2b.

Brittle compressive strength of seasonal pond ice

Compression experiments were performed on plates (152 x 152 x 25 mm^3) made from accretion pond ice collected during February, 2002 from the Lower Baker Pond (Wentworth-Orford, NH). The largest faces of the plates were parallel to the bottom free surface of the ice sheet (ice-water interface). Structurally, the ice was characterized by very large columnar grains and in general was transparent although it contained a moderate amount of gas bubbles.

Biaxial loading was applied monotonically in a plane parallel to the largest faces of the specimens. The minor (confining) stress σ_{22} was made to be proportional to the major compressive stress σ_{11}. The loading path, defined by the ratio $R = \sigma_{22} / \sigma_{11}$, was varied from $R = 0.0$ to $R = 0.14$. The strain-rate along the main compressive axis was $4.5 \pm 0.5 \times 10^{-3}\ s^{-1}$. All experiments were performed at $-10^{\circ} \pm 0.2^{\circ}$ C.

The results indicate that natural pond accretion ice was significantly weaker than laboratory S2 ice tested under identical conditions (2.9 MPa versus 4.5-5.0 MPa under uniaxial compression, and 4.5 MP versus 12 MPa under biaxial compression with $R = 0.14$).

The significantly lower strength is attributed primarily to the very large grains and to the presence of one or two major grain boundaries which acted as fault precursors. While this explanation is commonly valid in many materials, in the particular case of natural ice it is suggested that the presence of impurities, and in particular impurities segregated to the grain boundaries may have led to accelerated decohesion and increased

shear compliance of the boundaries and thus additional weakening of the specimens, compared to similar laboratory-grown ice.

The effect of impurities on ice

Creep of H_2SO_4-doped ice

Both high-purity ice single crystals and doped single crystals with H_2SO_4 concentrations between 70 p.p.b. and 170 p.p.b. were produced by unidirectional freezing of either distilled water or a solution of distilled water and sulfuric acid over a period of 7 to 10 days.

Tensile creep specimens with a gage section of 34 mm x 9 mm x 9 mm were fabricated from large single crystals. The basal planes were oriented at 45° ± 5° to the tensile axis. All creep test were performed at –20° ± 0.3°C under a constant load of 1.3 kg, which provided an initial stress of 0.16 MPa.

The experiments demonstrated that both undoped and the doped (150-170 p.p.b.) ice crystals were capable of undergoing large deformations with strains in excess of 200% (Figure 3a-b; for comparison an undeformed specimen is shown in Figure 3c). Figure 3d shows the typical ribbon-like appearance of the central section of the specimens after testing. While a consistent difference between the secondary creep rates of the doped and undoped specimens was not observed, a clear difference was observed in the length of the secondary creep regimes which lasted approximately three days in the doped crystals compared to about one and a half days for the undoped ones (Figure 3e).

The effect of impurities on recrystallization

Primary recrystallization and subsequent grain boundary migration was investigated using 10 mm x 10 mm x 30 mm cuboidal specimens cut from single crystals doped with either sulfuric acid (70 ppb to 170 ppb) or sodium chloride and magnesium sulfate (1ppm to 10 ppm). Large shear strains were imparted to the ice crystals by slowly pushing the specimens under constant load (1.7 Kg) through an ECAE jig with $2\phi = 120°$

at $-2° \pm 0.2°C$ over a period of four to six hours. The extrusion produced a shear strain of approximately 1. Prior to deformation the basal planes were oriented at 75°-90° to the direction of the applied load. After extrusion the specimens were "annealed" at the same temperature for up to 90 hours.

Figure 4 shows typical results from sulfuric acid-doped specimens. In the crystal on the left in Figure 4, which contained 70 p.p.b. sulfuric acid, new grains (marked A) nucleated in less than three hours after deformation. In contrast, recrystallization in the specimen on the right, which contained 170 p.p.b. H_2SO_4 only started after approximately 30 hours.

Subsequently, the new grain boundary in the 70 p.p.b.-doped specimen migrated through most of the length of the crystal, whereas over a comparable time period, the grain boundaries of the recrystallized grain in the 170 p.p.b.-doped specimen hardly moved. Thus, it is evident that the presence sulfuric acid in relatively small concentrations retards both recrystallization and grain growth in ice, i.e. the same effect as solutes in metals.

In contrast, NaCl and $MgSO_4$ do not have such a dramatic effect on recrystallization. Nucleation of new grains in the heavily deformed single crystals occurred up to concentrations of about 5ppm (Figure 5). The difference is attributed to the extra protons (H^+) supplied by the sulfuric acid which allow the hydrogen bonds to re-orient more readily, thus increasing the mobility of the dislocations. In the concentration range mentioned above (much less than a ppm) it is expected that the mobility would increase with the content of H_2SO_4 and consequently, following deformation, the more highly sulfuric acid-doped specimens undergo a recovery process rather than minimizing the stored strain energy through the creation of new stress-free grains such as in the case of NaCl and lightly-doped H_2SO_4 ice.

Measurements of the electrical conductivity of H_2SO_4-doped ice

Platinum electrodes were inserted (through the thickness of the sample) into polycrystalline H_2SO_4-doped (mostly 10 ppm) ice both into the grain boundary and in the lattice, and a 1 V d.c. potential was applied. Because of the low currents involved, it was

necessary to put the sample in an aluminum box to minimize the electrical noise. The data in Figure 6 are typical of the currents measured as a function of time in the ice lattice and through the grain boundary. The background noise is also shown. Note the large difference between the current in the bulk and the current through the grain boundary. With the exception of a few ripples, the bulk current was essentially constant at a little under 1 nA. In contrast, the current through the grain boundaries decreased from initial values of 8-14 nA to from 2-4 nA, and continued to vary ± 0.5 nA about these levels. The decrease in current through the grain boundary as a function of time presumably represents polarization of the electrodes. Although referred to as grain boundary current, the current shown in Figure 4 arises from the superposition of two components: (1) the actual current through the boundary which is the main component, and (2) the current through the adjacent lattice, which is much smaller. Interestingly, preliminary tests performed on ice with a much larger concentration of H_2SO_4 (1000 ppm) indicated that, given enough time (3-4 hours), the magnitude of the boundary current eventually decreases to levels comparable to the current in the lattice (although for 1000 ppm H_2SO_4 the current in the lattice is itself quite large).

Imaging of Un-Coated Snow Crystals Using a Scanning Electron Microscope

Our recently-developed SEM technique was used to image samples of fresh snow collected during or shortly after snowfalls from untouched regions on the ground in the immediate vicinity of the laboratory in Hanover, New Hampshire (February and March 2002).

In order to allow for an objective assessment of our technique, we compared the images of several types of crystal aggregates and individual snow crystals obtained without the application of a conductive coating with images of similar features obtained by Wergin and others[1] who used platinum coating on snow. The types of features

[1] Wergin W.P., Rango A., and Erbe E., 1995, Observations of Snow Crystals Using Low-Temperature Scanning Electron Microscopy, Scanning, Applications, Vol. 17, 41-49, and Wergin W.P., Rango A., Erbe E., and Murphy C.A., 1996b, Low Temperature SEM of Precipitated and Metamorphosed Snow Crystals Collected and Transported from Remote Sites, Journal of the Microscopy Society of America (JMSA), Vol. 2, No. 3, 99-112.

compared included individual or assemblies of flat hexagonal plates (Figure 7a-b), collection of short prismatic ice crystals held together by a matrix (Figure 7c-d), sinuous metamorphosed dendritic forms exhibiting bonding or sintering with the adjacent crystals (Figure 7e), and asymmetric crystals (Figure 7f). The comparative analysis of the images revealed that in the context of our procedure, the absence of a metallic coating, at least for the categories represented in Figure 7, did not result in a reduction of the level of detail compared to similar observations on coated snow crystals. Moreover, as mentioned above, the technique has the great advantage of allowing the use of EDS, as well as real-time observations of sublimation-induced morphological changes in the snow-aggregates. To illustrate the latter point, Figure 7c and Figure 7d show collections of prismatic crystals being slowly exposed as the matrix in which they are embedded (possible a frozen water droplet) sublimates away. Such observations would have not been possible had the ice been coated.

Impact for Science

Modeling the mechanical behavior of natural ice requires knowledge of the effects of impurities on the underlying physical processes. The effects of impurity particles on creep of ice[2] and on grain growth[3] have been studied but the effects of soluble impurities have received little attention. For natural ice, the latter case is of great importance since sulfuric acid, or salts such as magnesium sulfate and sodium chloride are frequently encountered impurities. Our work, which focuses primarily on the effects of sulfuric acid on recrystallization of ice, is among the first in this field. Soluble impurities and inclusions may play an important role in limiting the brittle strength of

[2] Budd, W.F. and Jacka, T.H., 1989. A review of ice rheology for ice-sheet modeling. Cold regions science and technology, 16 (2), 107-144.
Durham, W.B., Kirby, S.H., and Stern, L.A., 1992. Effects of dispersed particulates on the rheology of water-ice at planetary conditions. Journal of geophysical research-Planet, 92 (E12), 20883-20897.
Hooke, R.LeB., Dahlin B.B., and Kauper, M.T., 1972, Creep of ice containing dispersed fine sand. Journal of glaciology, 63, 327-346.
[3] Alley R.B., Perepezenko J.H., and Bentley C.R., 1986a, Grain-growth in polar ice: 1. Theory. Journal of Glaciology, 32 (112), 413-424.
Alley R.B., Perepezenko J.H., and Bentley C.R., 1986b, Grain-growth in polar ice: 2. Application. Journal of Glaciology, 32 (112), 425-433.

natural ice by reducing the cohesion of grain boundaries through segregation to such interfaces.

The technique we have developed for the study of snow without the application of a conductive metallic coating has allowed us to investigate real-time morphological transformation of the ice crystals, and importantly has allowed the direct detection of impurities in snow by EDS.

Technology Transfer

The SEM/EDS technique developed here is being used in a NSF-funded project on the effect of particles on the creep of ice at U.S.A.-C.R.R.E.L. (Dr. D. Cole - P.I.) to study the chemistry and microstructure of particles and the surrounding lattice in ice. The x-ray topography techniques that we have previously developed will also be used in that project, using a recently acquired rotating-anode x-ray set, to measure dislocation densities in ice.

Personnel Supported

During this period the following personnel were wholly or partially supported:

D. Cullen - Ph.D. candidate

D. Iliescu - Post-doctoral Fellow

I. Baker - P.I.

Daniel Cullen was awarded his doctorate in June, 2002.

Awards

Ian Baker was elected a Fellow of ASM International in November 2001 and a Fellow of The Institute of Materials, Minerals and Mining (U.K.) in October, 2002.

Publications and Presentations

The following papers were published or submitted for publication:

"The Chemistry of Grain Boundaries in Greenland Ice", D. Cullen and I. Baker, Journal of Glaciology, 46 (2001) 703-706.

"Observation of Impurities in Ice", D. Cullen and I. Baker, Microscopy Research and Technique, 55 (2001) 198-207.

"Preliminary Microstructural and Microchemical Observations of Pond and River Accretion Ice", D. Iliescu, D. Cullen and I. Baker, submitted to Cold Regions Science and Engineering, 35 (2002) 81-99.

"Sulfate Crystallites in Vostok Accretion Ice", D. Cullen and I. Baker, Materials Characterization, 48 (2002) 263-270.

"Examination of Dislocations in Ice", I. Baker, Crystal Growth and Design, **2** (2002) 127-134. *(Invited)*

"Sulfate Crystallites in Vostok Accretion Ice", D. Cullen and I. Baker, Materials Characterization, 5498 (2002) 263-270.

"The Effects of H_2SO_4 on the Mechanical Behavior of Ice Single Crystals", I. Baker, Y.L. Trickett, D. Iliescu and P.M.S. Pradhan, in Creep Deformation Fundamentals and Applications, Ed. - R.S. Mishra, J.C. Earthman and S.V. Raj, TMS, Warrendale, PA (2002) 85-94.

"Structure, Chemistry and Properties of Grain Boundaries in H_2SO_4-Doped Ice", D. Iliescu, D. Cullen, C. Muscat and I. Baker, Proceedings of Microscopy and Microanalysis 2002, 1544-45CD.

"SEM/EDS Studies of Impurities in Natural Ice ", D. Cullen, D. Iliescu and I. Baker, Proceedings of Microscopy and Microanalysis 2002, 1398-9CD.

"Scanning Electron Microscopy of Vostok Acrretion Ice", D. Cullen and I. Baker, Proceedings of Microscopy and Microanalysis 2002, 1546-7CD.

"Imaging of Uncoated Snow Crystals Using a Low-Vacuum Scanning Electron Microscope", D. Iliescu and I. Baker, Journal of Glaciology, 48(162) (2002)

"The Structure and Chemistry of 94m GISP2 ice", I. Baker and D. Cullen, Annals of Glaciology, 35 (2003).

"Recrystallization, Grain Boundary Chemistry and Properties of Sulfuric Acid-Doped Ice", D. Iliescu, X. Li and I. Baker, Canadian Journal of Physics, 81 (2003) 395-400.

"The microstructural location of impurities in ice", I. Baker, D. Cullen, and D. Iliescu, Canadian Journal of Physics, 81 (2003) 1-9.

Scanning Electron Microscopy of Natural Ice", I. Baker, D. Cullen, D. Iliescu and R. Obbard, Proceedings of the Mike Meshii Symposium, in press

The following presentations were made:

"Studies of Natural and Artificial Ice", Johns Hopkins University February 14th, 2001.

"The Microstructural Location of Impurities in Natural Ice", I. Baker, D. Cullen and D. Iliescu, ARO Workshop on Snow and Ice, Hanover, NH, 5th-7th March, 2001.

"Impurities in Ice Cores", D. Cullen and I. Baker, 31st Annual Arctic Workshop, University of Massachusetts - Amherst, MA, 22nd-24th March, 2001.

"Microanalysis of Impurities in Ice from GISP2, Greenland and Byrd Station, Antarctica", D. Cullen and I. Baker, poster at the 2001 Spring meeting of the American Geophysical Union, May 29th – June 2nd, Boston, MA.

"Dislocations in Ice", I. Baker, 13th American Conference on Crystal Growth and Epitaxy, Burlington, Vt, August 12th - 16th, 2001. *(Invited)*

"The Microstructural Location of Impurities in Ice Cores", D. Cullen, I. Baker, International Symposium on Ice Cores and Climate, Kangerlussuaq, Greenland, August 19th – 23rd, 2001.

"Studies of Natural and Artificial Ice", Northeastern University October 26th, 2001.
The Structure and Chemistry of 94m GISP2 ice, D. Cullen and I. Baker, poster at 2001 Fall TMS meeting, Indianapolis, IN, November 4th-8th, 2001.

"Scanning electron microscopy and X-ray topography of Vostok ice", D. Cullen and I. Baker, poster presented at the 2002 Spring meeting of the American Geophysical Union, May 28th – 31st, Washington,DC.

The microstructural location of impurities in ice from GISP2 and Byrd Station", I. Baker, D. Cullen and R. Obbard, 2002 Spring meeting of the American Geophysical Union, May 28th – 31st, Washington,DC.

"The Microstructural Location of Impurities in Natural Ice ", I. Baker, D. Cullen and D. Iliescu, International Conference on the Physics and Chemistry of Ice (PCI 2002), St. Johns, NewFoundland, Canada, 14th-19th July, 2002.

"Recrystallization, Grain Boundary Chemistry and Properties of Sulfuric Acid-Doped Ice", D. Iliescu, X. Li and I. Baker, International Conference on the Physics and Chemistry of Ice (PCI 2002), St. Johns, NewFoundland, Canada, 14th-19th July, 2002.

"Structure, Chemistry and Properties of Grain Boundaries in H_2SO_4-Doped Ice", D. Iliescu, D. Cullen, C. Muscat and I. Baker, poster at Microscopy and Microanalysis 2002, Québec City, Canada, August 4^{th}-8^{th}, 2002.

"SEM/EDS Studies of Impurities in Natural Ice ", D. Cullen, D. Iliescu and I. Baker, Microscopy and Microanalysis 2002, Québec City, Canada, August 4^{th}-8^{th}, 2002.

"Scanning Electron Microscopy of Vostok Accretion Ice", D. Cullen and I. Baker, poster at Microscopy and Microanalysis 2002, Québec City, Canada, August 4^{th}-8^{th}, 2002.

"The microstructural location of impurities in polar ice", I. Baker, D. Cullen and R. Obbard, 2002 Fall TMS meeting, Columbus, OH, October 6^{th}-10^{h}, 2002.

"Studies of Natural and Artificial Ice", McGill University, November 26^{th}, 2002.

Figure 1: Thin-section (~2 mm thick) showing the typical microstructure of the top of the accretion portion of seasonal ice cover over Baker pond. Note the long and straight grain boundaries.

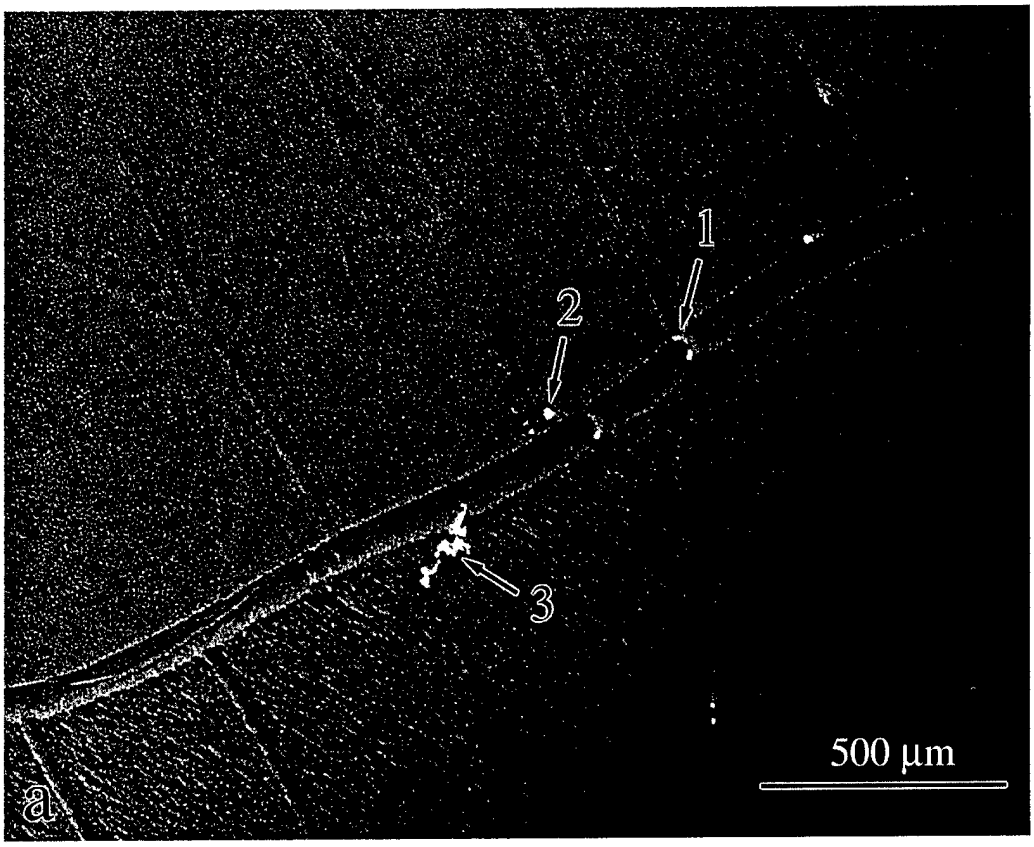

Figure 2: (a) SEM image showing that impurities in river ice segregate in small-size particles located within or on the edge of the grain boundary troughs; (b) The EDS spectra corresponding to the impurity aggregates marked in (a).

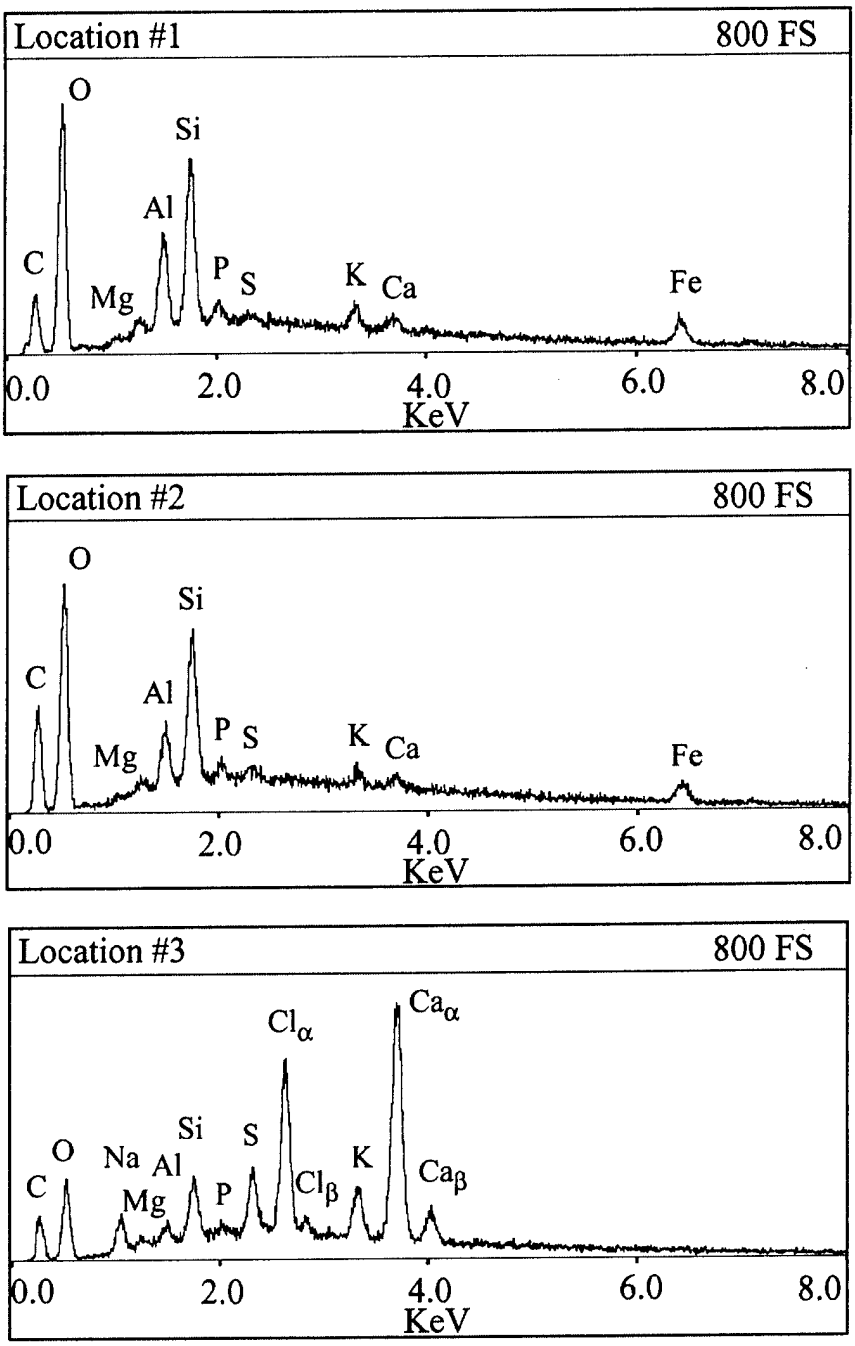

Figure 2: (a) SEM images showing that impurities in river ice segregate in small-size particles located within or on the edge of the grain boundary troughs; (b) The EDS spectra corresponding to the impurity aggregates marked in (a).

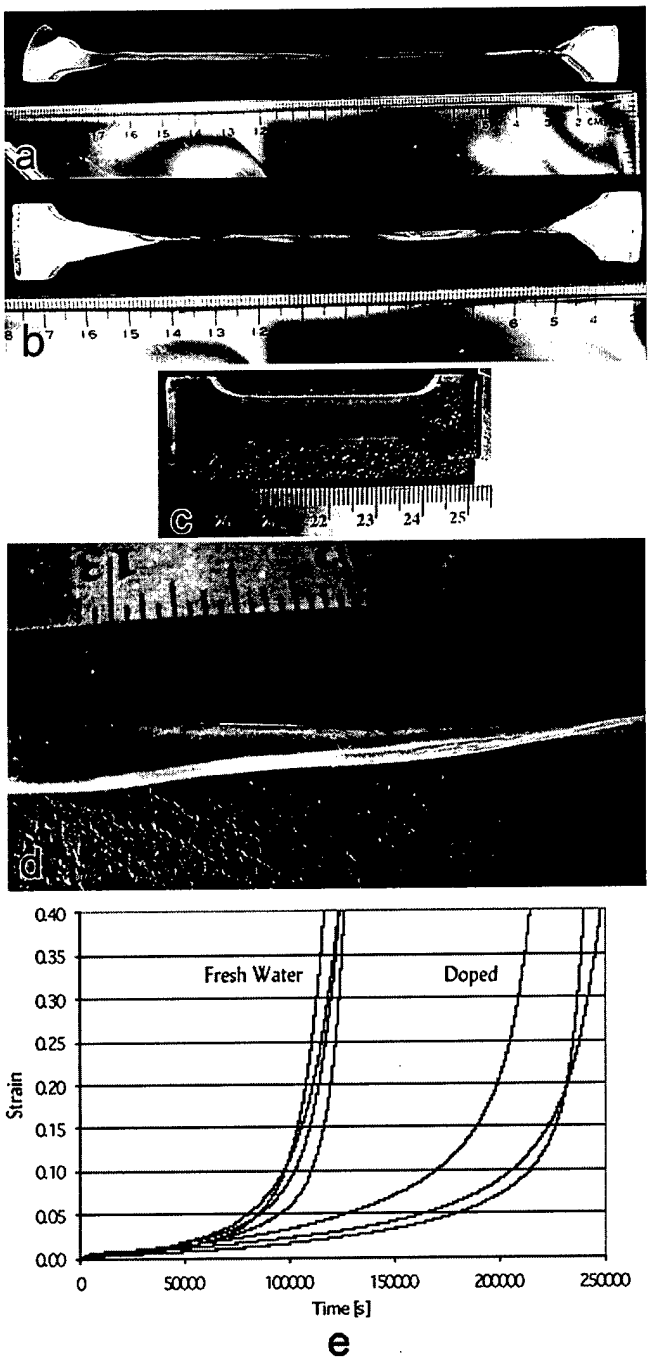

Figure 3: (a) Undoped and (b) sulfuric acid-doped single crystals subjected to creep under constant load at –20°C showing strains in excess of 200%; (c) undeformed specimen; (d) close-up of the central part of a sample similar to the ones shown in (a) and (b). Note the ribbon-like appearance; (e) strain versus time plots showing the longer secondary creep region for the doped crystals.

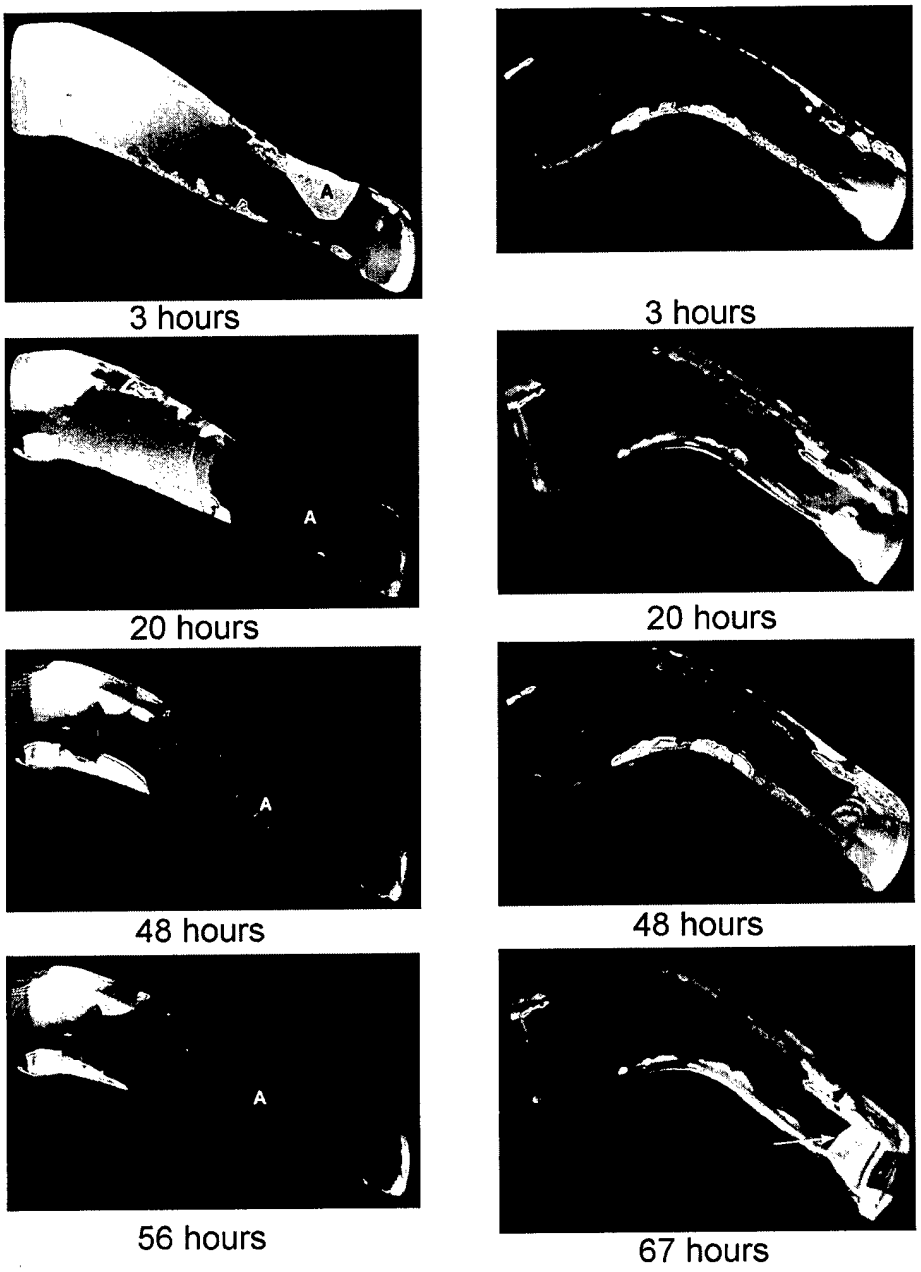

Figure 4: Photographs showing the effect of sulfuric acid on primary recrystallization and grain boundary migration after processing through an ECAE jig. The concentration of sulfuric acid was 70 ppb in the specimen on the left and 170 ppb in the specimen on the right. "A" marks a newly formed grain in the 70 ppb-doped specimen. The arrow (bottom right) points to an inclined grain boundary. Note the difference in appearance between the two crystals at comparable times and the much more rapid migration of the newly formed grain boundary in the less doped crystal (left).

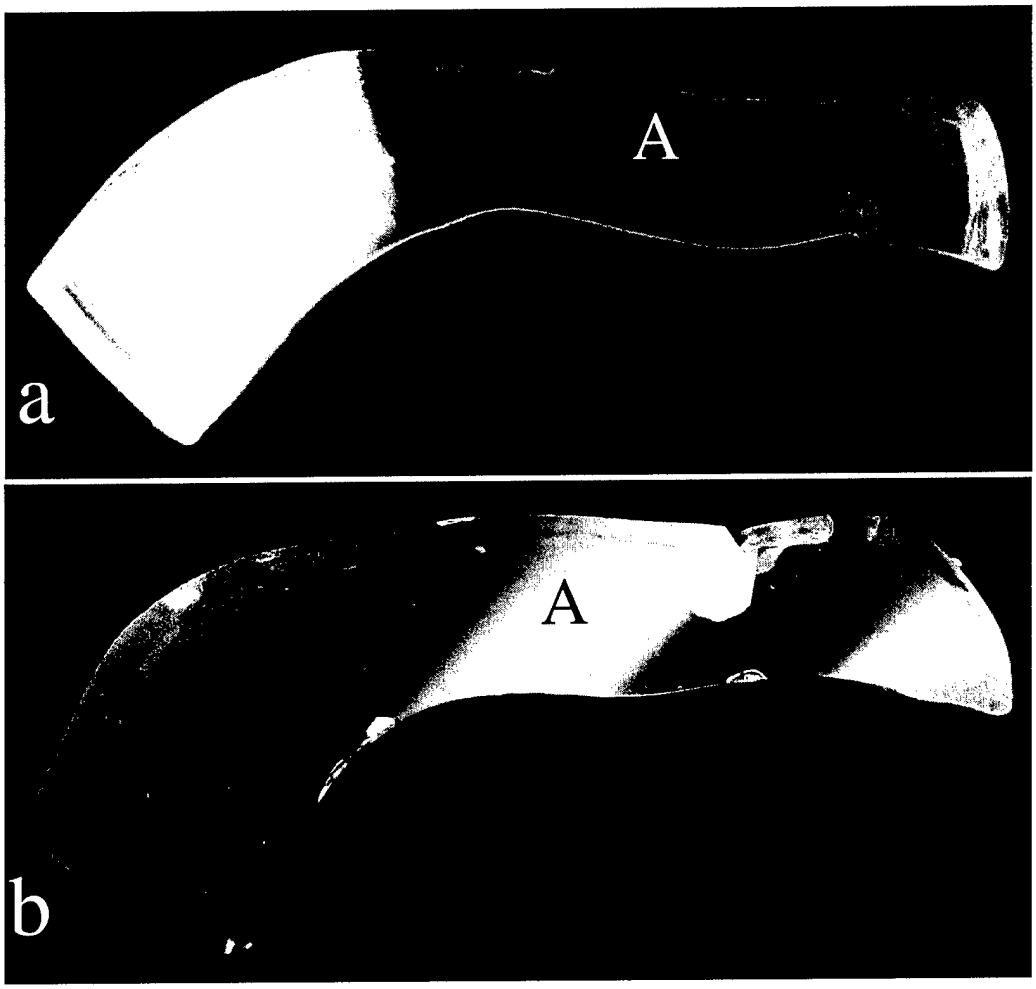

Figure 5: Photographs showing the effect of NaCl on primary recrystallization and subsequent grain boundary migration. Both specimens were extruded through an ECAE jig at –2°C. (a) specimen doped with about 2-3 ppm after 28 hours; (b) specimen doped with about 5 ppm after 25 hours. Concentrations in excees of about 5ppm seemed to significantly affect primary recrystallization and grain boundary movement (unlike sulfuric acid which virtually stopped recrystallization at about 150 ppb). Marked *A* are the newly formed grains.

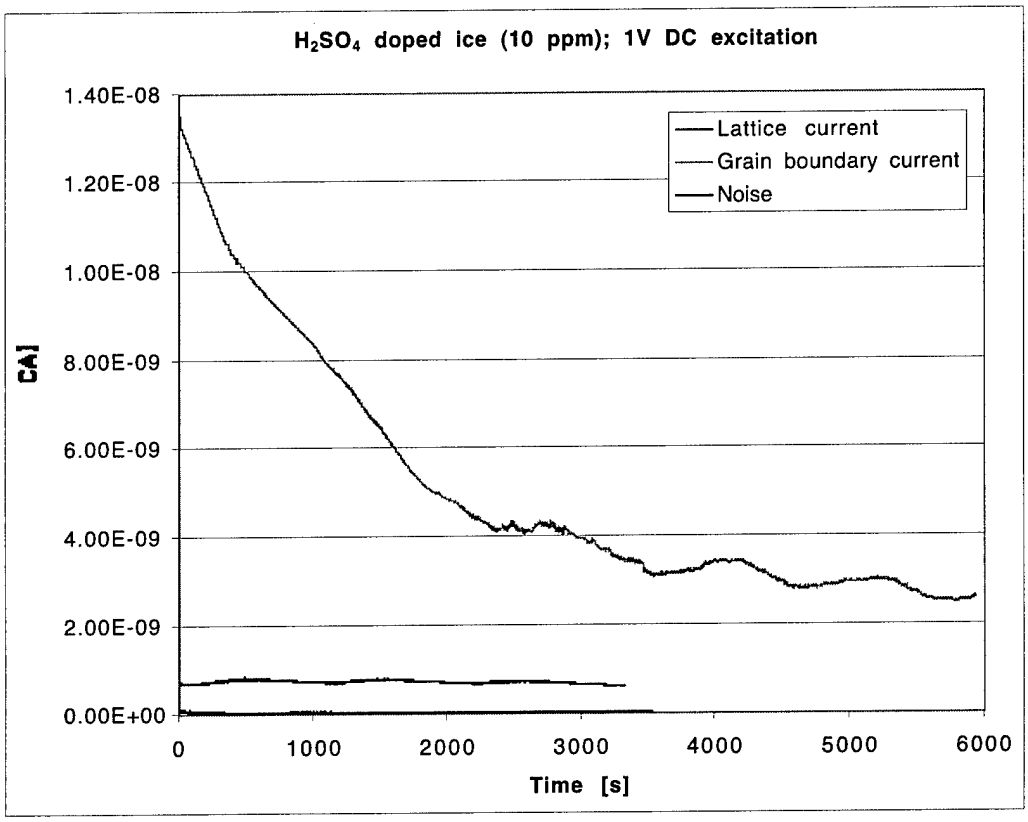

Figure 6: Typical current versus time curves for sulfuric acid-doped ice under 1V d.c excitation. Note the much larger conductivity of the grain boundaries compared to the grain interiors.

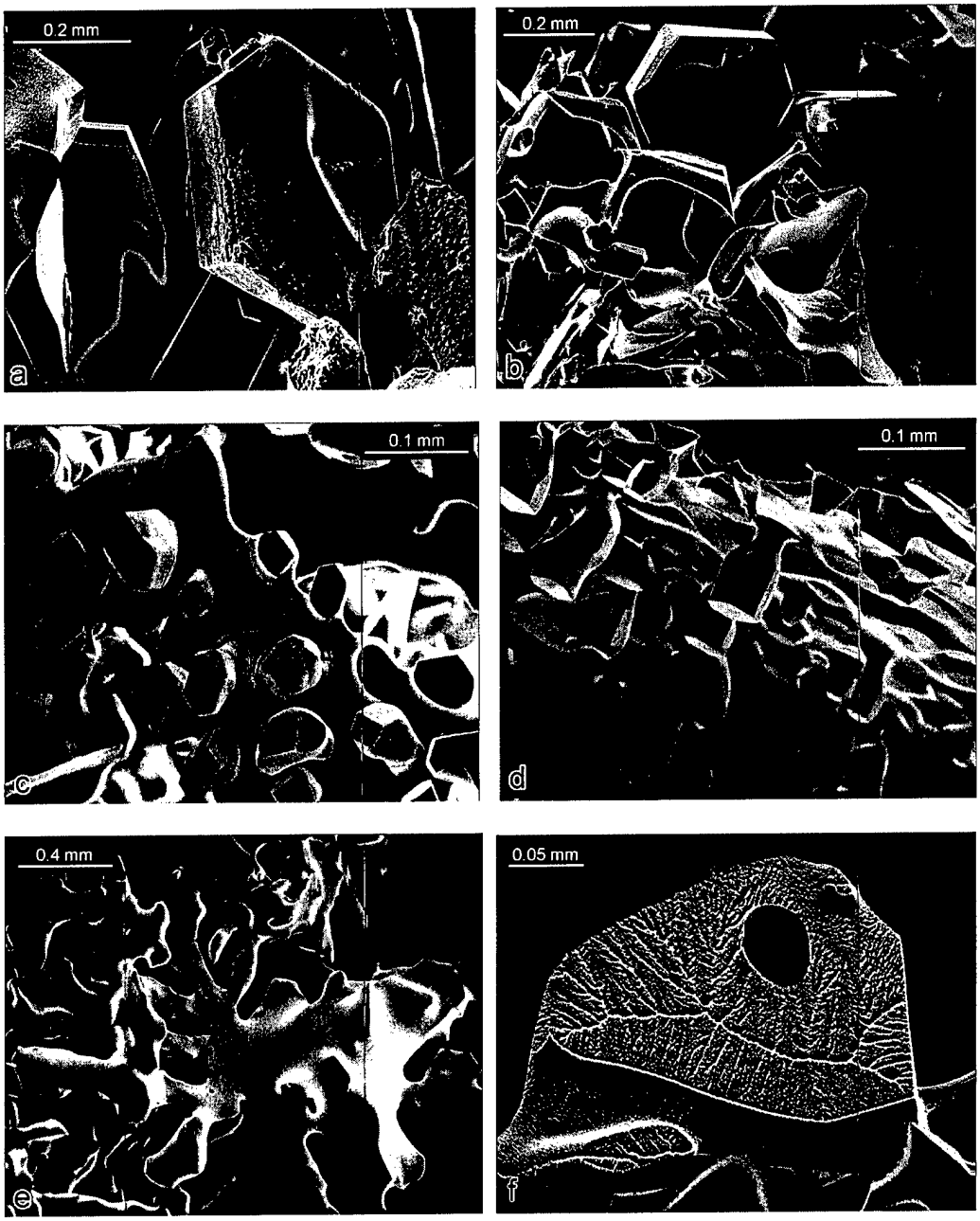

Figure 7: SEM images of four types of crystal aggregates and individual snow crystals obtained without a conductive coating. (a, b) Flat hexagonal plate crystals; (c, d) prismatic crystals embedded in a matrix; (e) rounded metamorphosed dendritic forms; (f) possible asymmetric dendritic features.